



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : C11D 11/02, 1/12, 1/37, 1/83, 17/00	A1	(11) International Publication Number: WO 99/10467 (43) International Publication Date: 4 March 1999 (04.03.99)
(21) International Application Number: PCT/GB98/02489 (22) International Filing Date: 19 August 1998 (19.08.98) (30) Priority Data: 9717723.2 21 August 1997 (21.08.97) GB (71) Applicant (for all designated States except US): IMPERIAL CHEMICAL INDUSTRIES PLC [GB/GB]; Imperial Chem- ical House, Millbank, London SW1P 3JF (GB). (72) Inventor; and (75) Inventor/Applicant (for US only): CLIGNET, Paul [BE/BE]; Rue de Renory 28, B-4102 Ougree (BE). (74) Agents: ROBERTS, Jonathan, Winstanley et al.; ICI Group Intellectual Property, P.O. Box 11, The Heath, Runcom, Cheshire WA7 4QE (GB).		(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, HR, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>
(54) Title: SYNTHETIC DETERGENT FORMULATIONS (57) Abstract Synthetic detergent formulations are made by spray drying an aqueous dispersion or emulsion of a synthetic detergent and a hydrophobic plasticiser and optionally a filler to produce a particulate product. In particular the synthetic detergent includes mild surfactants such as fatty isethionate or sulphosuccinate surfactants. By spray drying the emulsion or dispersion, a particularly uniform product is made which can readily be formed into synthetic detergent bars which may include soap to form combination bar products (combi-bars).		

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece	ML	Mali	TR	Turkey
BG	Bulgaria	HU	Hungary	MN	Mongolia	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MR	Mauritania	UA	Ukraine
BR	Brazil	IL	Israel	MW	Malawi	UG	Uganda
BY	Belarus	IS	Iceland	MX	Mexico	US	United States of America
CA	Canada	IT	Italy	NE	Niger	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NL	Netherlands	VN	Viet Nam
CG	Congo	KE	Kenya	NO	Norway	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NZ	New Zealand	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	PL	Poland		
CM	Cameroon	KR	Republic of Korea	PT	Portugal		
CN	China	KZ	Kazakstan	RO	Romania		
CU	Cuba	LC	Saint Lucia	RU	Russian Federation		
CZ	Czech Republic	LI	Liechtenstein	SD	Sudan		
DE	Germany	LK	Sri Lanka	SE	Sweden		
DK	Denmark	LR	Liberia	SG	Singapore		
EE	Estonia						

Synthetic Detergent Formulations

This invention relates to synthetic detergents and in particular to improved methods for the manufacture of products made with synthetic detergents.

In the manufacture of products made with synthetic detergent compounds, in particular the manufacture of bars using synthetic detergents ("syndet bars") and bars containing both synthetic detergents and soaps ("combination" or "combi" bars"), it is known that the compounded detergent formulations are difficult to form into bars. Currently, such bars are made by processes similar to those used for making bars from natural soaps (based on the salts, usually alkali metal salts of naturally occurring fatty acids), in particular by compounding the basic ingredients into flakes or extruded "noodles". Conventionally, these are made by mixing the raw materials using high shear blending of synthetic detergents, and in combi bars with soap(s), with a molten plasticiser in the presence of relatively small amounts of water. The result is a relatively inhomogeneous or macroscopic mixture. The mix is then flaked or extruded to give noodles. The bars are made from the flakes or noodles by working at moderately elevated temperatures using roll mixers and extruders (the processing in extruders is commonly referred to as "plodding"), followed by extrusion to form a slug of the compounded soap mixture which is then cut and pressed into the final bar form. A particular problem in the manufacture of syndet and combi bars is that the temperature range within which the plasticity of the formulation allows slug and bar manufacture is much narrower than is available in the manufacture of bars made from natural soaps. This necessitates relatively tight process control to make slugs and eventually bars which have adequate coherence to be of practical value. Even so, presently available syndet and combi bars are not fully satisfactory as products for domestic use. Further it is difficult to incorporate significant amounts of additives that provide useful properties in the final product as the additives typically cause changes in the plasticity profile of the overall mix sometimes causing softening and sometimes hardening, thus complicating processing.

This invention is based on the discovery that pre-processing a composition containing the synthetic detergent to produce a relatively finely divided powder or granulate can make subsequent processing much simpler, enabling the incorporation of additives more easily and at higher levels than is practical with conventional processing methods, and enabling the manufacture of products, particularly slugs to give cleansing bars which have superior uniformity and give better, particularly smoother, skin feel in use than conventional synthetic detergent cleansing bars.

Accordingly the present invention provides a method of making a formulated synthetic detergent product, which includes:

- 1 forming an aqueous dispersion or emulsion of a synthetic detergent and a hydrophobic plasticiser and optionally a filler; and
 - 2 spray drying the dispersion or emulsion,
- to produce a particulate product including the synthetic detergent and the hydrophobic plasticiser, and, optionally, the filler.

The invention includes a detergent material in particulate form which includes a synthetic detergent and a hydrophobic plasticiser and optionally a filler is a substantially homogeneous dispersion. In particular in this aspect of the invention, the detergent material is in particulate form including a synthetic detergent and an hydrophobic plasticiser, and, optionally, a filler, in substantially homogeneous dispersion, having a weight average particle size of from 150 to 1200 μm , particularly in free flowing non-dusting particulate form.

The invention further includes a method of making bars of synthetic detergent materials which comprises forming a synthetic detergent material in particulate form of or made by the spray drying method of the invention into bars.

The invention additionally includes a personal care synthetic detergent cleansing bar product which has been made from a synthetic detergent material in particulate form of or made by the spray drying method of the invention.

The synthetic detergent is (typically) an anionic or non-ionic surfactant. Suitable anionic types of surfactant include alkyl sulphates, such as lauryl, myristyl, stearyl and cetyl sulphates, alkyl sulphonates, alkyl ether sulphates, alkyl glycerol ether sulphonates, alkyl phosphate esters, ethoxylated alkyl phosphate esters, sarcosinates, taurate derivatives, alkyl sulphoacetates, hydroxyalkyl sulphonate esters, such as isethionate esters, particularly of fatty carboxylic acids, for example cocoyl isethionic acid, lauryl isethionic acid and stearyl isethionic acid, usually used as an alkali metal e.g. sodium salt, alkyl sulphosuccinates, such as di-sodium and/or potassium lauryl, oleyl and stearyl sulphosuccinates, alkyl ether sulphosuccinates, alkyl sulphosuccinamates, and acyl glutamates. Such anionic surfactants are usually used as metal, usually alkali metal especially sodium or potassium, ammonium, ethanolamine or alkali earth metal particularly magnesium salts.

The alkyl chains in such surfactants are typically C_9 to C_{20} , more usually C_{14} to C_{18} chains.

Suitable non-ionic types of surfactant include alkyl polysaccharides (more properly described as alkyl oligosaccharides) particularly where the saccharide residues are glucose residues and particularly where the alkyl groups are C₈ to C₁₆ alkyl groups, and especially lauryl or decyl glucoside, particularly having an average degree of polymerisation of from 1 to 2; sorbitan ester alkoxylates, particularly sorbitan laurate or stearate ethoxylates e.g. containing an average of from about 15 to about 30, particularly an average of about 20, ethylene oxide residues per molecule; glucamide surfactants such as glucamides of C₈ to C₂₂ acids; alcohol alkoxylate, particularly ethoxylate surfactants, especially C₁₀ to C₂₂ alkyl alkoxylates, particularly ethoxylates, typically containing 20 to 100 alkoxylate, particularly ethoxylate, residues; carboxylic acid ethoxylates especially of C₁₀ to C₂₂ carboxylic acids and containing 20 to 100 ethoxylate residues. The composition may also include ester surfactants such as glycerol mono-esters such as glyceryl mono-stearate, oleate or laurate, and citrate esters.

As is usual in personal care products, it is desirable to include mild surfactants. And it is for this reason that anionic surfactants where the hydrophile is the anionic residue of a relatively strong acid group, usually a sulphate or more usually a sulphonate group, are used in this invention.

Typical examples of such surfactants include carboxylic acids including fatty, particularly C₈ to 20 alkyl, esterified with hydroxyalkylsulphonic acids such as isethionic acid (2-hydroxyethane sulphonic acid; HO.CH₂.CH₂.SO₃H), as salts, usually alkali metal salts, particularly sodium salts of C₁₂ to C₂₀ carboxylic isethionates, especially sodium cocoylisethionate and sulposuccinate surfactants. Combinations of isethionate surfactants and sulposuccinate surfactants, for example approximately equal proportions of sodium cocoyl isethionate and sodium sulposuccinate, give particularly good results. Such mild surfactants can be used alone or in combination with other (more harsh) surfactants, but such mixtures will have intermediate mildness properties.

The use of such mild surfactants is an important feature of the invention such that it forms a specific part of the invention which accordingly includes a method of making a formulated synthetic detergent product, which comprises

- 1 forming an aqueous dispersion or emulsion of a surfactant composition comprising at least one fatty isethionate foaming surfactant, optionally but desirably at least one sulposuccinate salt; a hydrophobic plasticiser; and optionally a filler; and
 - 2 spray drying the dispersion or emulsion,
- to produce a particulate product including the synthetic detergent and the hydrophobic plasticiser, and, optionally, the filler.

In such mild surfactant based products, the surfactant is desirably mainly or predominantly a mild surfactant, in particular, at least 50%, desirably at least 75% and especially at least 90%, by weight of the surfactant is a mild surfactant, particularly an alkyl sulphonate and/or a sulposuccinate and/or a fatty isethionate. This forms a further specific aspect of the invention.

Other surfactants, particularly synthetic surfactants can be included in the synthetic detergent products of and made by the present invention. Suitable synthetic surfactants include amphoteric surfactants such as betaine derivatives, imidazoline derivatives, and fatty amphotoacetates, particularly coco-amphotoacetates. The composition may also include soaps, particularly fatty acid soaps, particularly alkali metal salt of fatty C_{12} to C_{20} carboxylic acids such as sodium and/or potassium stearate and/or myristate and/or cocoate. The amount of fatty acid soaps, when used, can vary widely. Relatively small amounts can be used to improve the cosmetic properties of syndet formulations such as foaming. In such applications the amount will usually be from 5 to 30% by weight based on the total amount of detergent in the composition. These levels are typical of some commercial combi-bar formulations. At such levels the soap may substitute for part of or add to the synthetic detergent in the formulation. Compositions containing relatively large amounts of soap can be considered as soap based compositions which include syndets to make them more mild or to make the base more compatible with additives. In such systems, the soap can be up to about 95%, typically 75 to 95%, by weight based on the total amount of detergent in the composition. Levels intermediate these ranges are used in combination formulations intended to have properties intermediate those of syndets and soaps. For such products the amount of soap is usually in the range 30 to 75%, more usually 40 to 60%, by weight based on the total amount of detergent in the composition.

When fatty acid soaps are included in the compositions, the manufacturing process for the bar products can involve including part or all of the soap in the emulsion which is spray dried, combining soap e.g. in the form of conventional noodles, flakes or pellets, with spray dried syndet powder or a combination of these.

The component described generally as a hydrophobic plasticiser serves the function of softening the detergent product to improve its workability, particularly in the manufacture of detergent bars. The manufacture of detergent bars is typically carried out at temperatures of from 35 to 50°C and the hydrophobic plasticisers used in the invention typically have melting or softening points from ambient temperature up to about 90°C, desirably up to 50°C. Suitable materials include fatty acids,

particularly C₁₂ to C₂₂ fatty acids such as stearic, myristic and coconut oil fatty acids; fatty alcohols, particularly C₁₄ to C₂₂ fatty alcohols such as stearyl alcohol; waxes such as paraffin wax or hydrogenated oils such as hydrogenated castor oil and jojoba wax (hydrogenated jojoba oil).

The filler is an optional but very desirable component of the detergent formulation used in the invention. It is a relatively inert finely divided particulate material, usually having an average particle size of less than 50 µm, typically from 2 to 25 µm which provides binding, additional plasticity and improved skinfeel properties. The filler can be an organic material such as starch or an inorganic material such as talc.

The relative (percentage) proportions by weight of the detergent and the hydrophobic plasticiser are usually as follows:

Material	broad range	usual range
detergent	55 - 80	60 - 75
hydrophobic plasticiser	45 - 20	40 - 25

When the filler is present the relative proportions of the detergent and the hydrophobic plasticiser are generally within the above ranges and the filler is added as from 2 to 25, particularly 8 to 22 and especially 10 to 20% of the three component system. This gives three component percentage compositions by weight typically as follows (including for clarity the no-filler proportions):

Filler range	amount (%)	broad range				usual range			
		detergent		hydrophobic plasticiser		detergent		hydrophobic plasticiser	
		from	to	from	to	from	to	from	to
	0	80	55	20	45	60	75	40	25
broad	5	76	52	19	43	57	71	38	24
	25	60	41	15	34	45	56	30	19
usual	8	73.5	50.5	18.5	41.5	55	69	37	23
	22	62.5	43	15.5	35	47	58.5	31	19.5
preferred	10	72	49.5	18	40.5	54	67.5	36	22.5
	20	64	44	16	36	48	60	32	20

Other components such as are conventionally included in syndet and combi bars can be included for example polymers such as high molecular weight polyethylene glycol (PEG), typically having a molecular weight of greater than 100kD and polysaccharides which improve binding, skinfeel and compatibility, conditioners such as polyquaternary ammonium compounds (polyquats) humectants

such as glycerol and sorbitol ethoxylates such as sorbeth-30, emollient oils such as mineral oils e.g. isoparaffin oils, natural or synthetic triglycerides, propoxylates alkyl ethers, perfumes, pigments and dyes. Such materials will usually be included in conventional concentrations, for each type of these components typically from 0.1 to 5% by weight of the overall formulation. Depending on their nature, they can be included at any suitable stage in manufacture, by inclusion in the emulsion (for non-volatile components), by addition to the spray dried product or by inclusion at later stages of processing.

The dispersion or emulsion in water can be made up by simply dispersing the individual components, detergent(s), hydrophobic plasticiser and optionally filler in water and warming and stirring the mixture to form the dispersion or emulsion. However, it is generally desirable to preheat the water to about or above the melting temperature of the hydrophobic plasticiser, a typical range is from about 75 to about 90°C, then to add the filler (if used) followed by the detergents and finally to add the hydrophobic plasticiser in liquid (molten) form and to stir the mixture. Generally high shear mixing is not needed to disperse or emulsify the components once the mixture has been made up. This makes the processing relatively simple and straightforward. The dispersion or emulsion is then spray dried typically by feeding the dispersion or emulsion to a spray drier using a heated stream of gas, typically air or nitrogen, usually as dry gas as the drying medium. The temperature of the dry gas will be high enough, and sufficiently above the dew point of the gas, to enable efficient removal of water i.e. to supply enough heat to evaporate the water present at the flow rates used, but not so high that it causes decomposition of the product. Typically, the dry gas temperature will be from 160 to 250°C.

After spray drying the powdered detergent composition can be subject to mild agglomeration e.g. in a fluidized bed, particularly with the recirculation of fines, particularly to reduce the proportion of fines and modestly to increase the average particle size. This aggregation reduces dusting and eases the incorporation of additives. The weight average particle size of the spray dried detergent product is generally in the range from 50 to 1200 µm. More usually, if the powder is not agglomerated, the weight average particle size is typically from 50 to 250 µm and if it is agglomerated, the weight average particle size (after agglomeration) is typically from about 200 to about 750 µm.

Bars can be made from the spray dried powder straightforwardly using conventional techniques (allowing for the form of the feedstock). Thus the powder can be charged to a mixer and appropriate amounts of water, perfume and other additives, if desired, such as oils, humectants e.g. glycerine, and plant extracts and be added and the composition mixed until the components are

uniformly distributed. We have found that this can be more quickly than with detergents in the form of flakes or noodles. Thus, with the product produced according to the invention, mixing can be done in about 1 minute, where with flakes or noodles it would take 3 to 4 minutes. This can significantly enhance the output of the mixers without requiring investment in larger scale equipment. Further, because extended mixing increases the extent to which the compositions become soft and sticky, the ability to operate with short mixing times is an additional benefit.

The mixture is then passed to a bar making line where it can be milled in a 2 or 3-roll mill to give a sheet with the desired plasticity and temperature and then processed in a simplex extruder (usually fitted with a coarse grid) to extrude the slug of soap. In the conventional manufacture of syndet and combi bars, the milling step, particularly during start up, usually involves much adjustment of the set up to make a satisfactory product. We have found that using the spray dried product made by this invention, the set up of the milling stage is made much easier. Alternatively, the processing on the 2/3-roll mixer can be omitted and the mixture passed directly to a plodder e.g. a duplex (2-step) plodder, and the soap bar extruded. The route chosen will depend on the desired degree of homogeneity in the product. The extruded slug can then be cut and stamped to form the soap bar product.

Using the spray dried material of this invention, we have found that after mixing, as described above, the product can be passed directly to the plodder or extruder. This contrasts with manufacture using conventional flakes or noodles because these materials usually require processing in a simplex extruder or plodder and/or roll-mill to homogenise the product and to give it a suitable plasticity before further plodding and bar extrusion. We have further found that the time needed for a processing line to settle to steady state operating conditions is much shorter using the spray dried material according to this invention. We believe that this is because the spray dried material provides better filling of the volume of the extruder and improved surface contact and higher internal friction of the material being extruded leads to a much quicker equilibration of the temperature.

It is an advantage of this invention that the generally adverse effects on processability (in bar making) of mixer additives is much less than in conventional processes. Thus, much higher amounts of water can be included before the material becomes too soft to process. For example, using conventional flakes noodles or pellets, the maximum amount of water that can be added is typically from 2 to 4%, but using powder made according to the invention amounts as high as 10 to 13% can be added without major difficulty. Also, additives, such as perfumes, particularly alcohol or hydroalcohol based perfumes, that have an adverse effect on plasticity and binding in conventional

systems sometimes making it impossible to extrude the detergent as a slug, have much reduced impact on the process carried out using the spray dried product according to this invention.

The invention includes a method of making a synthetic detergent bar which includes the steps of:

- 1 forming an aqueous dispersion or emulsion of a synthetic detergent and a hydrophobic plasticiser and optionally a filler; and
 - 2 spray drying the dispersion or emulsion,
- to produce a particulate product including the synthetic detergent and the hydrophobic plasticiser, and, optionally, the filler;
- and subsequently forming the particulate product including the synthetic detergent into a bar.

In particular the synthetic detergent bar can be made by mixing optional further components (if any) with the particulate product including the synthetic detergent followed by milling in a 2 or 3-roll mill and subsequent processing in a simplex extruder (usually fitted with a coarse grid) to extrude the slug of soap which is subsequently cut and stamped into bars. Alternatively, the processing on the 2/3-roll mixer can be omitted and the mixture passed directly to a plodder e.g. a duplex (2-step) plodder, and the soap extruded as a slug which is subsequently cut and stamped into bars.

The following Examples illustrate the invention. All parts and percentages are by weight unless otherwise stated.

Materials Used

Arlatone SCI	sodium cocoyl isethionate ex ICI Surfactants
Radiasurf 7140/3	glycerol mono stearate ex Oleofina
Radiacid 0427	stearic acid ex Oleofina
Talc de Luzenac	finely divided talc as filler ex Luzenac
Perfumes	Blue Water hydroalcoholic perfume ex Dragoco
Zetasap 813A	syndet base ex Zschimmer & Schwarz containing: 50% (w/w) anionic surfactant: disodium C12 to C18 sulfosuccinate and sodium alcohol C12 to C18 sulfate 23% fillers including corn starch 23% plasticizers including cetearyl alcohol and paraffin 3.5% water 0.1% Titanium dioxide white to ivory-coloured noodle-shaped pellets.
Zetasap 5165	syndet base ex Zschimmer & Schwarz containing: 40% (w/w) anionic surfactant: disodium C12 to C18 sulfosuccinate and sodium cocoyl isethionate 26% fillers including corn starch 30% plasticizers including cetearyl alcohol glyceryl stearate and paraffin 3.5% water 0.1% Titanium dioxide white to ivory-coloured noodle-shaped pellets.
Tensianol LSA-LM	syndet base ex ICI Surfactants which contains: 43% anionic surfactants disodium lauryl sulphosuccinate and sodium cocoyl isethionate 20% refatting oil phase cetearyl alcohol and hydrogenated castor oil 20% corn starch and minor amounts of cocamidopropyl betaine, glycerine, mineral oil, titanium dioxide, and water

The commercial syndet base materials are manufactured by blending the base components at relatively low water content (less than 50% of the total composition) and evaporative drying followed by extrusion as noodles, flaking or pelleting. Those skilled in the art recognise that such products, particularly those in noodle or pellet form require high shear processing, typically using a roll mill to give the product the acceptable plasticity and surface to accept additives before it is extruded as bars. Without this sort of processing, the product does not extrude successfully.

Example 1

A synthetic detergent formulation was made up containing the following components:

material	weight %	role
Arlatone SCI	14.28	surfactant
Radiasurf 7140/3	11.22	hydrophobic plasticiser
Radiacid 0427	2.0	
Talc de Luzenac	1.5	filler
water	70	dispersion medium

The dispersion was made by heating the water to 80°C and dispersing the Arlatone SCI under moderate stirring until homogeneous. The Radiasurf 7140/3 and Radiacid 0427 were heated to 80°C and added to the aqueous mix under stirring until it was homogeneously dispersed. The dispersed mix was held at 80°C under moderate stirring until further processing by spray drying. The resulting emulsion was spray dried using a NIRO pilot dryer P63 at an evaporation rate in the range 10 to 60 kg water.hour⁻¹. The spray drying was carried out under the following conditions: temperatures: slurry at spray drier inlet 75°C; drying chamber inlet (using hot air as the drying medium) 195 to 200°C; outlet 90°C; air flow 400 m³.hour⁻¹. The resulting powder had an average particle size of less than 200 µm and a water content of 3%.

This material was converted into bars using a pilot single screw extruder at ambient temperature. Adding 10% of water did not adversely influence the plasticity of the slug or the hardness of the resulting cleansing bars.

Example 2

A spray dried product similar to that of Example 1 was made by spray drying an aqueous emulsion formed by dispersing Zetasap 813A at 30% in water preheated to 80°C with moderate stirring. This temperature is above the melting temperature of the blend of the hydrophobic plasticiser components of the syndet formulation. The resulting emulsion was spray dried using a NIRO pilot dryer P63 at an evaporation rate in the range 10 to 60 kg water.hour⁻¹. The spray drying was carried out under the following conditions: temperatures: slurry at spray drier inlet 75°C; drying

chamber inlet (using hot air as the drying medium) 195 to 200°C; outlet 90°C; air flow 400 m³.hour⁻¹. The resulting powder had an average particle size of less than 200 µm and a water content of 2.5%.

Example 3

Example 2 was repeated except that Zetasap 5165 was used instead of the Zetasap 813A used in Example 2. The resulting powder had a water content of 2.5% (the original Zetasap 5165 has a water content of between 4 and 7%) by weight.

The resulting powder, which was very similar in physical form to that of Example 2, was converted directly at ambient temperature in a simple single screw pilot scale laboratory extruder with addition of 10 to 15% of water into syndet slugs which can be stamped into bars without making the extruded slug or stamped bars soft. In contrast, the commercial flake material needs to be roll-milled or heated to 40°C and recycled many times in the extruder to mimic the roll-mill plasticising step and to allow incorporation of the water/glycerin processing additives (2.5%).

The powder syndet material (heated or at ambient temperature) was mixed with the additives and water/glycerin process aids until homogeneous, adding material to the extruder gradually and extruding it into noodles using a 4mm diameter grid at the extrusion head. The body of the extruder was cooled at this stage to avoid slipping. The syndet noodles were subsequently passed through the extruder a second time to mimic the sequential use of simplex and duplex extruders. For the second pass a heated extruder head was used to make a rectangular (cross section) slug. After extrusion, the slug was cut and stamped into bars.

Example 4

Tensiolin LSA-LM flake syndet base was dispersed at 30% by weight in water at 80°C under moderate stirring. This temperature is above the melting temperature of the hydrophobic plasticiser components of the syndet formulation and enables it to be emulsified in the surfactant solution formed on dispersion. Subsequently, 2000 l of this emulsion was spray dried in a Niro MSD spray drier having a water removal capacity of about 1000 kg water.hour⁻¹. A vibrating fluidised bed and filter were placed at the bottom of the dryer to granulate the spray dried material and to separate and recirculate small particles to the dryer. Hot air was used as the drying medium and the drying chamber inlet temperature was 180°C, the drying chamber outlet temperature was 65°C and the airflow temperature in the fluidised bed was 75 to 85°C. The resulting free flowing dust free powder had a particle size of between 300 and 1200µm and a water content of 2.5% (Tensiolin LSA-LM flake has a water content between 2 and 6%) by weight.

This powder product was further processed on an industrial line. In comparison with the original flake form of the syndet, the mixing time reduced by 50% and the absorption capacity of the syndet was increased. Thus using 100 kg of powder, 8 kg water, 3 kg of an hydroalcoholic perfume and 1 kg almond oil were added and all these additives were readily absorbed. This was accomplished without an adverse effect on the end properties of the syndet bar products. It is well understood in the art that this cannot be achieved using conventional flake, noodle or pellet materials.

We found that it was not necessary to use a roll mill to process the powdered syndet, some of the powder was processed using a roll mill to see whether the processability of the powder in a roll mill was satisfactory. (This allows for the practical situation where a bar manufacturer has a roll mill in the production line and it would be inconvenient to remove it just to process powdered syndet material.) The powdered syndet was processed through a roll mill without difficulties or further readjustment.

Generally it is well understood in the art that it is not practical to operate so simply using conventional flake, noodle or pellet syndets. Using flakes, noodles or pellets, it is usually necessary to heat the roll mills on start up and to use a relatively wide gap between the rolls to avoid the roll mill blocking. After start up, the roll mill gaps needs to be narrowed so to enable the syndet to be processed to give the desired plasticity and to avoid leaving hard fragments in the milled syndet. Further, frictional heating means that the roll mill need to be cooled to avoid sticking and blockage. All this needs careful handling and coordination until steady state operating conditions are reached.

The product was evaluated in use by skilled personnel and the touch and skinfeel of bar made from the powdered syndet was judged to be much better than otherwise similar bars made from conventional flaked syndet.

Example 5

It is well known that perfumes, especially alcohol and in some cases hydroalcoholic based perfumes tend to change the binding and plasticity properties of syndet bases. The following tests show the superiority of the material made according to the invention compared with flaked material. The powdered syndet made as described above, was converted into bars using a simple - single screw pilot extruder Example 5a and 5b samples). Samples of the same syndet formulation but in the form of flakes (as supplied) were also converted into bars on the same equipment (Comparative Example C5a and C5b samples). In one test, ethanol was used as an additive to simulate an alcohol based perfume and in the other Blue Water perfume was used.

The syndet material (heated or at ambient temperature) was mixed with the additives and process aids and supplied to the extruder and extruded in noodles using a 4mm diameter grid at the extrusion head. The amount (percentage based on the detergent composition weight) of water that

could be added during mixing to improve processing without it becoming sticky was noted and is given as Water% in Table 1. For the powdered material it was satisfactory to use an inlet temperature of 22°C, but for the flaked material it was necessary to warm it so that the temperature of the material at the inlet was 40°C to enable satisfactory processing through the extruder. (The body of the extruder was cooled at this stage to avoid slipping.) The plasticity of the extruded noodles was assessed and is indicated in Table 1 as Noodle plasticity. The material was subsequently passed through the extruder a second time to mimic the sequential use of simplex and duplex extruders on a full scale production line and a heated extrusion head was used to make a rectangular (cross section) slug which, after extrusion, was cut and stamped into bars. If this did not give a mix with adequate plasticity for processing into bars, the mix was again processed through the extruder making three passes in all. In Table 1 the number of Passes =1 indicates that satisfactory plasticity was achieved after 1 extruder pass starting from extruded noodles and Passes=2+ indicates that 2 passes were needed - not always giving a fully satisfactory result even then. The appearance and binding of the extruded slug were noted and are included briefly in Table 1. Binding = "poor" means that the cooled and matured bar breaks under low stress and "good" means it meets standard specification, breaks under high stress. Good plasticity is necessary to be able to stamp bars from the extruded slug at an economical rate and to obtain bars that do not "open" or "crack" when cooling.

Table 1

Sample	C5a	5a	C5b	5b
Additives - Material	ethanol	ethanol	Blue Water	Blue Water
- Amount (%)	3	5	3	3
Water (%)	2	8	none	10
Inlet temperature (°C)	40	22	40	22
Noodle plasticity	medium very soft and sticky	high	medium very soft and sticky	high
Passes	2+	1	2+	1
Slug - Appearance	soft tends to break (poorly bound)	hard very plastic	soft tends to break (poorly bound)	hard very plastic
- Binding	poor	good	poor	good

Claims

- 1 A method of making a formulated synthetic detergent product, which includes:
 - 1 forming an aqueous dispersion or emulsion of a synthetic detergent and a hydrophobic plasticiser and optionally a filler; and
 - 2 spray drying the dispersion or emulsion, to produce a particulate product including the synthetic detergent and the hydrophobic plasticiser, and, optionally, the filler.
- 2 A method as claimed in claim 1 wherein the synthetic detergent is or includes at least one fatty isethionate foaming surfactant and, optionally, at least one sulphosuccinate salt;
- 2 A method as claimed in either claim 1 or claim 2 wherein the synthetic detergent includes one or more anionic surfactant from alkyl sulphates, alkyl sulphonates, alkyl ether sulphates, alkyl glycerol ether sulphonates, alkyl phosphate esters, ethoxylated alkyl phosphate esters, sarcosinates, taurate derivatives, alkyl sulphoacetates, hydroxyalkyl sulphonate esters, alkyl sulphosuccinates, alkyl ether sulphosuccinates, alkyl sulphosuccinamates, and acyl glutamates.
- 3 A method as claimed in claim 3 wherein the anionic surfactant is present as an alkali metal, ammonium, ethanolamine or alkali earth metal salt.
- 4 A method as claimed in any one of claims 1 to 3 wherein alkyl chains in the anionic surfactant are C₉ to C₂₀ chains.
- 5 A method as claimed in any one of claims 1 to 4 wherein the synthetic detergent includes one or more non-ionic surfactant from alkyl polysaccharides; sorbitan ester alkoxylates; glucamide surfactants; alcohol alkoxylate surfactants; and carboxylic acid ethoxylates.
- 6 A method as claimed in any one of claims 1 to 5 wherein the synthetic detergent includes at least 50% by weight of the surfactant of an alkyl sulphonate and/or a sulphosuccinate and/or a fatty isethionate mild surfactant.
- 7 A method as claimed in any one of claims 1 to 6 wherein the synthetic detergent includes at least one betaine derivative, imidazoline derivative, or fatty amphoteric surfactants.

- 15 -

- 8 A method as claimed in any one of claims 1 to 7 wherein the emulsion additionally includes one or more alkali metal salt of a fatty C₁₂ to C₂₀ carboxylic acid.
- 9 A method as claimed in any one of claims 1 to 8 wherein the hydrophobic plasticiser is one or more C₁₂ to C₂₂ fatty acid; C₁₄ to C₂₂ fatty alcohol; paraffin wax; or hydrogenated castor oil or jojoba wax.
- 10 A method as claimed in any one of claims 1 to 9 wherein the filler is an inert finely divided particulate material having an average particle size of less than 50 µm.
- 11 A method as claimed in claim 10 wherein the filler is starch or talc.
- 12 A method as claimed in any one of claims 1 to 11 wherein the amount of detergent is from 55 to 80% by weight and the amount of hydrophobic plasticiser is from 20 to 45% by weight both based on the combined weights of detergent and hydrophobic plasticiser.
- 13 A method as claimed in any one of claims 1 to 11 wherein the amount of detergent is from 41 to 76% by weight the amount of hydrophobic plasticiser is from 19 to 34% by weight and the amount of filler is from 5 to 25% by weight based on the combined weights of these three components.
- 14 A detergent material which includes a synthetic detergent, a hydrophobic plasticiser and, optionally, a filler, in substantially homogeneous dispersion and having a weight average particle size of from 150 to 1200 µm, in free flowing non-dusting particulate form.
- 15 A method of making bars of synthetic detergent materials which comprises forming a synthetic detergent material in particulate form as claimed in claim 14 or made by the method claimed in any one of claims 1 to 13.
- 16 A personal care synthetic detergent cleansing bar product which has been made from a synthetic detergent material in particulate form as claimed in claim 14 or made by the method claimed in any one of claims 1 to 13.

INTERNATIONAL SEARCH REPORT

In **ational Application No**
PCT/GB 98/02489

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C11D11/02 C11D1/12 C11D1/37 C11D1/83 C11D17/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	GB 845 376 A (THOMAS HEDLEY & CO. LTD.) 24 August 1960 see page 2, line 98 - page 3, line 17; claims; examples 1-3 ---	1-5, 9-11, 15-17
A	US 4 111 853 A (SHULTZ ANDREW ET AL) 5 September 1978 see column 8, line 38 - line 53; claim 1 ---	1,3-5,15
A	GB 2 231 579 A (UNILEVER PLC) 21 November 1990 see page 6, line 2 - page 7, line 12; claims; examples ---	1,3,6,8, 9,15
A	US 4 612 136 A (NOVAKOVIC MARIO ET AL) 16 September 1986 see claims ---	1,2, 15-17
	-/-	

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search

17 November 1998

Date of mailing of the international search report

01/12/1998

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Grittern, A

INTERNATIONAL SEARCH REPORT

Int lional Application No

PCT/GB 98/02489

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication where appropriate, of the relevant passages	Relevant to claim No.
A	<p> DATABASE WPI Section Ch, Week 7406 Derwent Publications Ltd., London, GB; Class D25, AN 74-10485V XP002084726 & JP 49 002321 B (AJINOMOTO KK) , 19 January 1974 see abstract ----- </p>	<p>1, 3-5, 14-17</p>

INTERNATIONAL SEARCH REPORT

Information on patent family members

In itional Application No

PCT/GB 98/02489

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
GB 845376 A		BE 573819 A FR 1211197 A NL 234111 A	14-03-1960
US 4111853 A	05-09-1978	NONE	
GB 2231579 A	21-11-1990	NONE	
US 4612136 A	16-09-1986	US 4696767 A	29-09-1987